United States
Environmental Protection
Agency

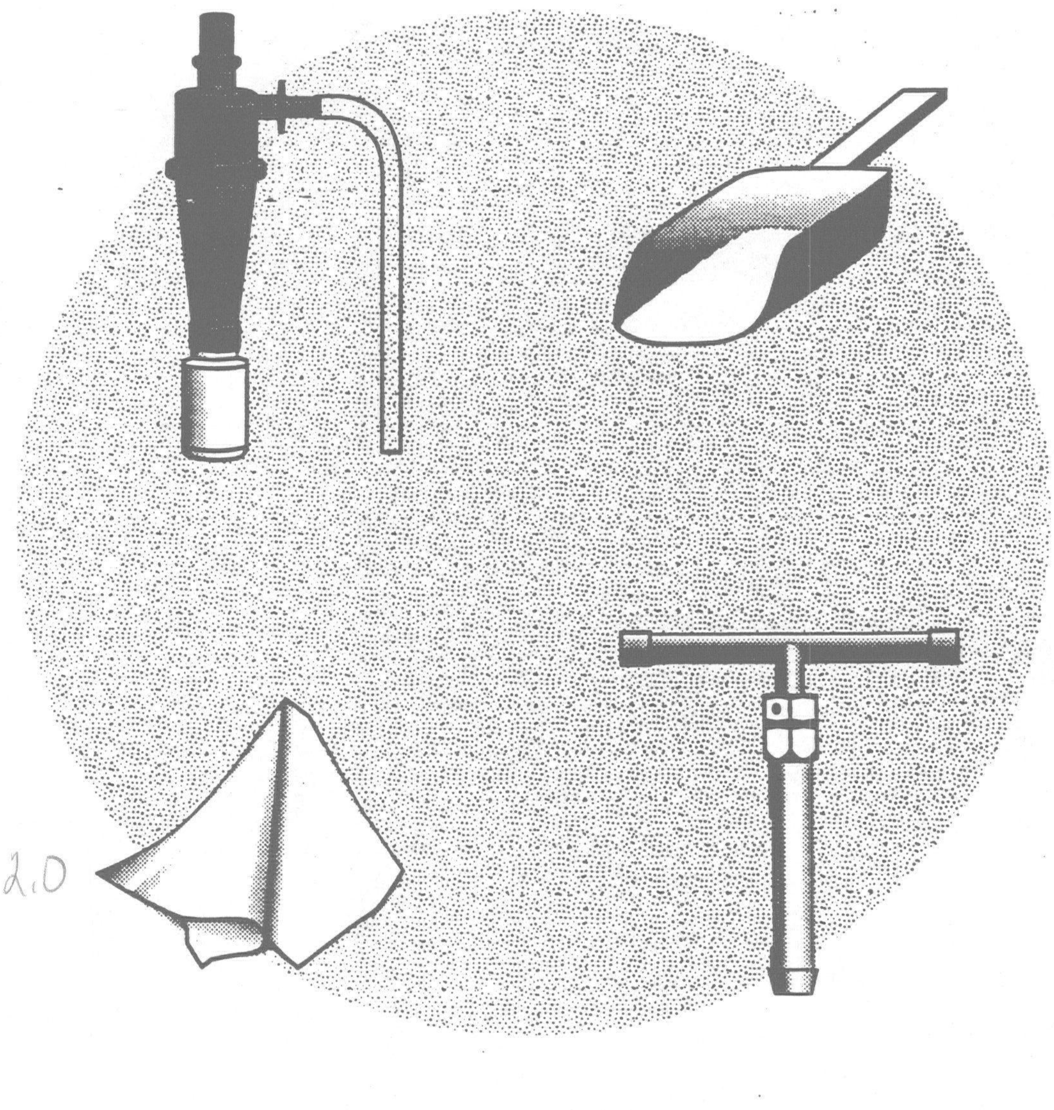
Pollution Prevention
and Toxics
(7404)

EPA 747-R-95-001
March 1995

EPA

Residential Sampling for Lead: Protocols for Dust and Soil Sampling

Final Report



LIBRARY
US EPA Region 4
AFC/9th FL Tower
61 Forsyth St. S.W.
Atlanta, GA 30303-3104

Residential Sampling for Lead: Protocols for Dust and Soil Sampling

Final Report

For U.S. Environmental Protection Agency
Office of Pollution Prevention and Toxics
Technical Programs Branch, MC-7407
Northeast Mall, Room G012
401 M Street, S.W.
Washington, D.C. 20460

Attn: Darlene Watford

Work Assignment 4-10(02)
MRI Project No. 9803

March 29, 1995

Disclaimer

This document has been reviewed and approved for publication by the Office of Prevention and Toxics, U.S. Environmental Protection Agency. The use of trade names or commercial products does not constitute Agency endorsement or recommendations for use.

Table of Contents

A. Introduction 1
- 1.0 Overview 1
- 2.0 Purpose 1
- 3.0 Related Documents 1
- 4.0 Number and Location of Samples 2
- 5.0 Sample Collection 2
- 6.0 Laboratory Analysis 3

B. Protocol for Collection of Soil Samples for Lead Determination 6
- 1.0 Introduction 6
- 2.0 Equipment and Supplies 6
- 3.0 Sampling Procedure 8
- 4.0 Quality Control 11
- 5.0 Glossary 13

C. Protocol for Collection of Dust Samples for Lead Determination Using Wipe Sampling 15
- 1.0 Introduction 15
- 2.0 Equipment and Supplies 15
- 3.0 Sampling Procedure 16
- 4.0 Quality Control 20
- 5.0 Glossary 23

D. Protocol for Collection of Dust Samples for Lead Determination Using Vacuum Sampling 25
- 1.0 Introduction 25
- 2.0 Equipment and Supplies 25
- 3.0 Sampling Procedure 27
- 4.0 Quality Control 33
- 5.0 Glossary 36

E. References 38

A. Introduction

1.0 Overview

This document provides guidance for the collection of soil and settled dust samples for subsequent determination of lead. Collection of soil samples is performed using either coring or spooning methods. Collection of settled dust samples are performed using wipe or vacuum methods. Advantages and disadvantages of each method are discussed. Protocols presented in this document are capable of producing samples for lead determination results in µg per gram of soil, µg per gram of dust, and µg per ft^2 of dust.

2.0 Purpose

The purpose of this document is to provide detailed sampling procedures to maintain uniformity in data collection of lead in soil and settled dust samples. Analytical results obtained from samples collected using these sampling procedures will eventually be used for comparison to numerical health-based standards developed by the U.S. Environmental Protection Agency (EPA) for discerning unsafe lead levels, according to Title X, Section 403: *Identification of Dangerous Levels of Lead.*

Today, a variety of approaches to sample collection for lead determinations are being utilized. Substantial resources are applied to the generation of lead data. The use of standardized protocols should improve the value of lead data gathered by a wide variety of organizations and individuals.

3.0 Related Documents

Currently, EPA is developing health-based standards for lead in soil and house dust under the Toxic Substances Control Act (TSCA), Title X, Section 403. Furthermore, EPA will soon publish standards on the conduct of residential lead risk assessments for the purposes of training risk assessors (TSCA, Title IV, Section 402). Both standards are interwoven and extremely important in addressing public health issues regarding lead.

For guidance to identify, control, or abate lead hazards in housing, consult the U.S. Department of Housing and Urban Development document, Guidelines for the Evaluation and Reduction of Lead-Based Paint Hazards in Housing (HUD, 1994).

To obtain more information on lead, call the National Lead Information Center Clearinghouse at (800) 424-LEAD. In the Washington, D.C., area, call (202) 833-1071.

Protocols presented in this document are equivalent to ASTM methods[1-2] for the collection of soil and dust.

4.0 Number and Location of Samples

A sampling plan is needed to determine where and how many samples are to be collected at any given sampling site (or housing area). Sampling plans typically include a variety of sampling locations, including collection of soil samples from near building locations (drip-line locations) and away from building locations, collection of dust samples from painted friction surfaces (such as window areas), and other high traffic areas. A complete sampling plan is developed under a specific lead determination objective and should be made prior to any sample collection. Specific sampling plan guidance is provided in the EPA document Residential Sampling for Lead: A Process for Risk Assessment, 1994.

5.0 Sample Collection

The amount of lead in soil and settled dust samples can be expressed as a lead loading (weight of lead per area sampled, typically $\mu g/ft^2$), or lead concentration (weight of lead per weight of sample, typically µg/g). Soil samples are generally reported in concentration results, and dust samples can be reported using both measures. Currently, more data are available relating blood lead levels to dust loading than to lead dust concentration. However, since there are limits in the amount of dust and soil material that can be ingested (or inhaled) by an individual, a lead value that incorporates the amount of dust, such as lead concentration, may provide important information for accurate evaluation of lead hazards. Unfortunately, technical difficulties exist for collection of dust with the intent to report lead results in concentration units. These difficulties are discussed in this document. In summary, the collection method should be selected according to the desired lead measurement unit because all collection methods do not produce results both in lead loading and concentration terms.

The collection protocol for soil samples is presented in Section B. The protocol, which provides for either scoop or coring type sampling options, focuses on collection of the top 0.5 inches of soil surface. It is assumed that the top 0.5 inches represents the portion most likely to cause an immediate hazard to occupants.

Two collection protocols are presented for lead in settled dust: wipe dust and vacuum dust collection. These protocols are presented in Sections C and D. Both protocols are intended for collection of settled dust as opposed to airborne particulate. Settled dust is generally referred to as dust material in the range of 5 to 500 µm in diameter. However, these collection protocols are not necessarily limited to collection of material in this range. Wipe dust collection is limited to reporting lead results in loading terms

($\mu g/ft^2$). Vacuum dust collection can generate both lead loading and lead concentration results. However, vacuum dust collection is generally more difficult and costly to perform than wipe dust collection.

6.0 Laboratory Analysis

Samples collected for lead determination are generally analyzed using atomic spectrometry methods. These instrumental methods require samples to be converted from a solid to a liquid form prior to lead measurement. This conversion process is commonly referred to as sample preparation. Sample preparation generally includes several initial handling steps, followed by a digestion process, that solubilize the lead contained in the sample. The accuracy and precision of the lead determination is dependent on both the sample preparation and instrumental analysis activities. An estimate of accuracy and precision can be obtained by submitting Quality Control (QC) samples to the laboratory together with field samples. Preparation of appropriate QC samples is discussed in the sample collection protocols presented in this document. Although this document is not intended as a guide on laboratory analysis procedures, a brief discussion of sample preparation and instrumental analysis activities, as they relate to the collection of soil and settled dust sample, is presented below.

6.1 Sample Preparation of Soil Samples

A number of laboratory sample preparation methods can be used for the preparation of soil samples. Hot plate digestions, such as SW846 method 3050[3] or ASTM ES 36-94[4], utilize nitric acid (sometimes with hydrochloric acid) and hydrogen peroxide for oxidation of sample components and solubilization of lead. Microwave digestions, such as 3051[3], utilize nitric acid for oxidation of sample components and solubilization of lead. Sample size limits exist for both digestion methodologies. Samples collected in the field and submitted to the laboratory are generally much larger than the digestion methods are capable of accommodating. Therefore, a subsample of the original soil sample must be used for the lead determination. This requires the use of a homogenization process prior to digestion to generate a representative subsample. The water content of collected soils can be widely variable. Therefore, to provide consistency for lead data comparisons under variable soil and weather conditions, soil samples must be reported on a dry weight basis. This requires inclusion of a drying process into sample preparation method used for soil analysis.

In summary, any sample preparation method used for lead determination in soil samples must include a homogenization and drying process prior to subsampling for digestion and solubilization of the lead.

6.2 Sample Preparation of Wipe Dust Samples

Wipe dust samples can be prepared in a manner similar to soil samples (i.e., digestion using acids and hydrogen peroxide). However, microwave digestion methods are not generally applicable because the large bulk of the wipe material used to collect the samples interferes with the digestion process. Modifications to standard hot plate digestion methods, such as SW846 method 3050[3], are generally required to assure that the wipe samples are completely covered with the digestion reagents (acids and hydrogen peroxide) during the digestion process. The ASTM dust wipe digestion, ES 36-94[4], is a modification of SW846 method 3050[3]. Unlike soil samples, wipe dust samples cannot be subsampled; therefore, no homogenization procedures are required for preparation of these samples.

In summary, any sample preparation method used for lead determination in wipe dust samples must include sufficient reagent volumes to completely digest both the wipe itself and the collected dust.

6.3 Sample Preparation of Vacuum Dust Samples

Vacuum dust samples also can be prepared in a manner similar to soil samples (i.e., hot plate or microwave digestion using acids or hydrogen peroxide, or both). However, if these samples are intended for reporting lead results in concentration units (µg/g) then the total sample weight must be determined, and the sample preparation procedure must include a specific sample weighing procedure. This reporting option requires a determination of the total collected sample weight. This determination is performed by inclusion of gravimetric procedures before field sampling (called prefield gravimetrics) and after field sampling (called postfield gravimetrics). Both prefield and postfield gravimetrics must be performed under identical humidity and temperature conditions to help minimize errors caused by the hydroscopic nature of the sample media (filters or filter cassettes). Similar to wipe dust samples, vacuum dust samples cannot be subsampled; therefore, no homogenization procedures are required for preparation of these samples.

In summary, any sample preparation method used for lead determination in vacuum dust samples that are intended for reporting lead results in concentration units (µg/g) must include prefield and postfield weighing procedures, quantitative transfer procedures, and digestion procedures.

6.4 Instrumental Measurement

Lead measurements in digested soil and dust samples can be performed using a variety of instrumental techniques. Atomic spectrometry methods commonly used include Flame Atomic Absorption Spectrometry (FAAS), Graphite Furnace Atomic

Absorption Spectrometry (GFAAS), and Inductively Coupled Plasma Atomic Emission Spectrometry (ICPAES). A variety of methods covering the use of these techniques, such as SW846 methods 7420[3], 7421[3] and 6010[3], or ASTM E 1613-94[5], can be used for lead measurements. In general, FAAS and ICPAES both have sufficient detection capability for lead determinations in all soil samples and most dust samples. However, for clean environments, GFAAS detection, which has approximately a 10-fold improvement in detection capability over FAAS and ICPAES, may be more appropriate for some dust samples. Lead measurement data must incorporate sample preparation variables, such as sample weights and digestion volumes, prior to reporting lead results.

B. Protocol for Collection of Soil Samples for Lead Determination

1.0 Introduction

This protocol provides for the collection of soil samples using either scooping or coring methods. The protocol is applicable for collection of soil samples for lead determination.

2.0 Equipment and Supplies

2.1 Scoop Sampling Equipment

2.1.1 Plastic centrifuge tube, 50-mL with screw-on cap. Used for scoop sampling or soil collection containers.

2.1.2 Sample collection container, resealable plastic bags (1 quart or 1 gallon) or sealable rigid-walled container with 50-mL minimum volume.

2.1.3 Spoon, plastic or stainless steel. Used for scoop sampling.

2.1.4 Steel or plastic measuring tape or ruler, divisions to at least $^1/_8$ inch.

2.2 Core Sampling Equipment

2.2.1 Coring probe, 0.5 inch minimum diameter, lead-free. The probe must be capable of being forced into hard ground to a depth of at least 2 inches without being damaged and have a mechanism to remove the core from the probe to permit discarding all but the top 0.5 inch of the soil core (see subsection 2.2.2). A number of devices can be utilized as a coring probe. Examples include: plastic or steel pipe, small tree sapling planters, and a professional stainless steel coring probe equipped with plastic liners, cross T-bar, and hammer.

2.2.2 Coring plungers, one with and one without a stop, sized to fit coring probe, lead-free. Removal of the soil core is generally performed using a pair of plungers machined to fit the inside diameter of the coring device. One plunger is equipped with a stop that limits extension of the plunger to within 0.5 inch from the far end of the coring probe. It is used to remove all except the top 0.5 inch of the soil core from the coring probe. The other plunger (without a stop) is used to remove the remaining 0.5 inch of the soil core from the coring probe.

2.2.3 Sample collection container, resealable plastic bags (1 quart or 1 gallon) or sealable rigid walled container with 50-mL minimum volume. If plastic bags are used, samples should be double bagged to protect against breakage and potential sample loss.

2.2.4 Spoon, plastic or stainless steel. Used for scoop sampling.

2.2.5 Steel or plastic measuring tape or ruler, divisions to at least $^1/_8$ inch.

2.3 General Supplies

2.3.1 Field notebooks, bound with individually numbered pages, see subsection 4.1.

2.3.2 Indelible ink marker, black or blue.

2.3.3 Ink pens, black or blue.

2.3.4 Packaging tape, used for sealing shipping containers.

2.3.5 Plastic bags, trash bags with ties.

2.3.6 Plastic gloves, powderless. Gloves with powder should not be used to avoid potential contamination of samples from powder material.

2.3.7 Preprinted field forms, preprinted with sufficient entry lines to address documentation needs presented in subsection 4.1

2.3.8 Shipping containers, cardboard or plastic for interim storage and shipment of sample collection containers.

2.4. Cleaning Supplies

2.4.1 Water, drinking water. Drinking water is used to assist in cleaning sampling equipment for soil sample collection. High purity water is not required for cleaning of sampling equipment because action levels for lead in soils are relatively high with respect to lead levels in drinking water.

2.4.2 Wipe, Disposable towelette moistened with a wetting agent. Used for cleaning sampling equipment. Wipe brands or sources should contain insignificant background lead levels. Laboratory analysis of replicate blank wipes should be used to determine background lead levels prior to use in the field. It is recommended to avoid brands of wipes that contain aloe because wipes containing aloe have been found to contain higher background lead levels. Background lead levels less than 10 µg per wipe are considered insignificant for most soil sampling activities.

3.0 Sampling Procedure

Two types of collection procedures are described in this section: scoop sampling and core sampling. Either procedure can be used for the collection of soil samples for lead determinations. Advantages and disadvantages of each are presented at the beginning of each procedure.

3.1 Scoop Sampling Procedures

Two procedures are provided for collection of soils at a given sampling location using a scooping methods. Scooping procedures are effective for collection from semisoft, sticky, and loose, sandy soils. Scooping procedures are not recommended for hard or frozen soils. Scooping procedures are less effective than coring methods for collection of multiple samples having uniform surface area sampled and consistent sampling depths. The scooping methods described here may result in collection bias toward increased amounts of surface soil as opposed to subsurface soil caused by the curvature of the scooping tools. Coring methods are generally free from this collection bias.

3.1.1 <u>Scoop Sampling Using a Plastic Centrifuge Tube</u>

3.1.1.1 Label a new plastic 50-mL centrifuge tube for use as a sample collection container (See subsections 2.1.2 and 5.5).

3.1.1.2 Pull on a pair of clean, powderless, plastic gloves. Gloves are used to protect the workers' hands and the integrity of the samples (to aid in avoiding cross-contamination between samples).

3.1.1.3 Using a measuring tape and a spare plastic 50-mL centrifuge tube, determine the proper scooping depth of the tube needed to collect approximately the top 0.5 inch soil. For example, if the plastic centrifuge tube is about 1 inch in diameter, then the proper scooping depth is to insert the tube into the soil until the soil surface is about even with the center of the tube.

3.1.1.4 Remove the cap of the plastic centrifuge tube and insert the open end of the tube into the soil to the desired depth as determined in step 3.1.1.3. Collect the soil into the tube by pushing or pulling the tube through the soil surface while maintaining the scooping depth of the tube (0.5 inch) in the soil. Move the tube a distance of 6-12 inches across the soil surface to complete collection of the soil into the tube. The movement of the tube across the sample location will result in a composite type soil sample.

3.1.1.5 Remove the tube from the ground, and wipe off any excess soil clinging to the outside of the tube and cap threads with a gloved finger. Replace the cap. Label the plastic centrifuge tube with sufficient information to uniquely identify the sample. Discard any gloves used during sample collection in a trash bag.

3.1.2 Scoop Sampling Using a Spoon

3.1.2.1 Label a new resealable plastic bag for use as a sample collection container (See subsection 5.5).

3.1.2.2 Pull on a pair of clean, powderless, plastic gloves. Gloves are used to protect the workers' hands and the integrity of the samples (to aid in avoiding cross-contamination between samples).

3.1.2.3 Using a measuring tape and a clean spoon, dig a small test hole adjacent to the sampling location to the depth of 0.5 inch. Use this hole as a visual aid during soil collection to help limit collection to a depth of 0.5 inch. Clean the spoon using a wipe until soil is no longer visible on the spoon.

3.1.2.4 Scoop the soil with the spoon down to the depth indicated by the test hole and place the sample in a sample collection container. Continue to collect soil until a circular hole of approximately 2 inch (0.5 inch deep) has been created.

3.1.2.4 Collect soil from two more locations within a 1 foot diameter circle around the first sample location, using the same procedure described above (subsections 3.1.2.2 through 3.1.2.4). Composite these scoop samples into the same sample collection container and seal the container in a manner that will minimize the air contained in the container. Discard any gloves used during collection in a trash bag after all three scoop samples have been collected and composited.

3.1.2.5 Pull on a pair of clean, powderless, plastic gloves. Clean the spoon using wipes and water until soil is no longer visible on the spoon. Discard any wipes and gloves used during cleaning in a trash bag. An alternative approach to cleaning is to use disposable spoons.

3.2 Core Sampling Procedures

The collection of soils using a coring method at a given sample location is provided in this subsection. Coring methods are effective for collection of soils from dense, hard, or sticky soils. Coring methods are not recommended for loose, sandy soils. Coring methods generally produce samples with more uniform surface areas and consistent sampling depths than scooping methods.

3.2.1 Label a new resealable plastic bag for use as a sample collection container (See section 5.5).

3.2.2 Pull on a pair of clean, powderless, plastic gloves. Gloves are used to protect the workers' hands and the integrity of the samples (to aid in avoiding cross-contamination between samples).

3.2.3 If needed, clean the coring probe and coring plungers using wipes or water. The sampling equipment is considered clean if no soil or other debris is visible on any of the surfaces. Check the stop on the coring plunger, equipped with a stop, to ensure that the plunger tip stops at a distance of 0.5 inch from the end of the coring probe. Adjust the stop if needed.

3.2.4 Place a directional arrow on the outside of the coring probe with the arrow head pointed toward the ground. This arrow identifies the orientation of the soil core with respect to the surface of the ground. The arrow is used to avoid inadvertent loss of the top of the soil core when the plunger is used to remove and collect the soil sample. If the coring probe is a professional stainless steel coring tool equipped with plastic liners, place the arrow on the outside of the plastic liner and orient the liner in the probe so that the arrow head is pointed toward the ground.

3.2.5 Grip the coring tool firmly between two hands and, using a slight twisting motion, drive the tool into the soil surface at the designated sampling location to a depth of at least 2 inches. The directional arrow (from section 3.2.4) must be pointing down. For extremely hard soils (i.e., hard packed or frozen), a hammer or other similar device may be needed to drive the tool into the ground. If conditions do not allow for full penetration to a minimum of 2 inches, make every effort to penetrate to a depth of at least 0.5 inches. If penetration is less than 0.5 inches, the documentation generated for the sample should indicate the approximate depth achieved.

3.2.6 Twist and snap the coring tool to one side and carefully remove the tool from the ground while retaining the soil core in the tool.

3.2.7 Insert a clean plunger, equipped with stop, into the top end of the coring probe or liner. (The bottom end is indicated by the arrow head drawn on the tool.

The top end is the opposite opening.) Push out all but 0.5 inch of the soil from the probe with the plunger. Using a gloved finger, wipe off the excess soil protruding from the probe. Allow the soil pushed out of the probe to fall on the ground near but not on the sampling location.

3.2.8 Using a clean plunger (without stop), push the remaining 0.5 inch section of the sample core into a sample collection container.

3.2.9 Collect two more soil cores within a 1 ft diameter circle around the first sampling location, using the same procedure described above (subsections 3.2.2 through 3.2.8). Composite these cores into the same sample collection container and seal the container in a manner that minimizes the air contained in the container. Discard any gloves used during collection in a trash bag after all three core samples have been collected and composited.

3.2.10 Pull on a pair of clean, powderless, plastic gloves. Clean the coring probe, coring plungers, and plastic inserts (if used) using wipes and water until soil is no longer visible on the equipment. Discard any wipes and gloves used during cleaning in a trash bag.

4.0 Quality Control

Adherence to quality control (QC) procedures is an important part of field sample collection. QC procedures, including documentation requirements, field QC samples, reference material check samples, and contamination avoidance are presented in this section.

4.1 Documentation

All field data related to sample collection must be documented. A field notebook or sample log form can be used to record field collection data. It is recommended that both types of documentation records (field notebooks and preprinted sample log forms) be utilized to assure collection of all relevant field data. Field data entries on documentation records must adhere to the following requirements:

4.1.1 General Documentation Requirements:

- All entries must be made using ink.
- Each page (notebook or form) must include the name of the person making the entries and the date of entries found on the page.
- Any entry errors must be corrected by using only a single line through the incorrect entry (no scratch outs) accompanied by the initials of the person making the correction and the date of correction.

- An initial page that correlates initials to a specific name must be generated and maintained with field data records to trace any initials used in notebooks and on data forms.

4.1.2 Specific Sampling Site Documentation Requirements:

- General sampling site description.
- Project or client name, address, and city/state location.
- Information as to what specific collection protocol was used.
- Information as to the use of interim storage and sample shipment mechanisms.

4.1.3 Documentation Required for Each Sample Collected:

- An individual and unique sample identifier and date of collection. This must be recorded on the sample container in addition to the field data records (notebook or form).
- Name of person collecting the sample and specific sampling location data from which the sample was removed.

4.2 QC Samples

4.2.1 Blank Samples. Normally, blank samples should be periodically collected (designated) throughout the sampling day at each sampling site. Field blank samples are used to identify any potential systematic lead contamination present in the sampling media and handling of samples during field collection and laboratory analysis activities. However, because soil samples are not collected on a sampling media such as a wipe of filter, there is no practical method for collection of a blank sample.

Although sampling equipment rinses can be used to collect potential field contamination related information, difficulties exist in laboratory processing of these "field blanks" with soil samples. Sampling equipment rinse-type field blanks cannot be carried through the homogenization/drying steps that are commonly applied to soil samples. In addition, lead results from equipment rinses are reporting in weight-volume units (i.e., µg/mL) and cannot be directly compared or related to the reported soil lead weight-weight results (i.e., µg/g). Therefore, no field blanks are recommended for soil sampling. Contamination effects should be minimized through adherence to the procedures specified in this protocol.

4.2.2 Blind Reference Material Samples. Reference materials should be periodically submitted to the laboratory for analysis as a check on adherence to proper laboratory sample preparation and instrumental analysis methods. Prepare a blind reference material by placing a portion (1-2 grams) of a reference material into a labeled sample collection container. It is recommended that the frequency of these QC samples be at least 1 per 20 field samples. Reference materials from NIST[6], such as SRMs 2709, 2711, and 2704, are readily available and can be used for preparing blind reference materials. Other sources of materials with known lead levels, such as soil materials from the ELPAT[7] program, may also be used as blind reference materials.

4.3 Contamination Avoidance

The following work practices should be followed to prevent cross-contamination of samples:

- Avoid tracking soil from one location to another by:
  - identifying and clearly marking all sampling locations upon arrival at the sampling site, and
  - instructing field team members to avoid walking through or over any of the marked sampling location areas.
- Use a new pair of powderless gloves at each sampling location.
- Inspect all sampling equipment for cleanliness prior to collection of each sample. Always clean suspect equipment if in doubt.
- Do not open sample containers until needed to collect each sample.
- When using bulk packed wipes, at each sampling location, discard the first two wipes pulled from the wipe container.

5.0 Glossary

5.1 Digestion, Sample preparation process that solubilizes lead present in the sample. The digestion process produces an acidified, aqueous solution called the digestate. A lead determination is made on the digestate during an instrumental measurement process.

5.2 Field Data, Any information collected at the sampling site.

5.3 Field Sample, Physical material taken from the sampling site that is targeted for lead determination.

5.4 Reference Material, Material of known composition containing a known amount of lead. These materials have typically been subjected to a large number of lead determinations to develop a lead result known to a high degree of confidence.

5.5 Sample Collection Container, Container for holding and transporting the samples from the field to the laboratory. The internal volume of the container must be sufficient to hold the entire collected sample.

5.6 Sampling Location, Specified area within a sampling site that is subjected to sample collection. Multiple sampling locations are commonly designated for a single sampling site. An example would be at the bottom of a specific slide in a specific playground area.

5.7 Sampling Site, Local geographical area that contains the sampling locations. A sampling site is generally limited to an area that can be easily covered on foot. An example would be John Smith's house at 3102 Nowhere Avenue, Detroit, MI.

5.8 Sample Preparation, Process used to ready a sample received from the field for lead determination using instrumental measurement methods. The process is dependent on the sample type and can include a large number of steps such as homogenization, drying, splitting, weighing, digestion, dilution to a final known volume, and filtering.

C. Protocol for Collection of Dust Samples for Lead Determination Using Wipe Sampling

1.0 Introduction

This protocol provides for the collection of settled dust samples from hard, relatively smooth, nonporous surfaces using wipe methods. The protocol is not applicable for the collection of settled dust samples from highly textured surfaces, such as brickwork and rough concrete, and soft fibrous surfaces, such as upholstery and carpeting. The protocol is capable of producing samples for lead determination results in loading terms (μg/ft^2).

2.0 Equipment and Supplies

2.1 Sampling Equipment

2.1.1 Disposable shoe covers (optional), see subsection 4.3.

2.1.2 Masking tape, used for holding down sampling templates and marking sampling locations.

2.1.3 Sample collection container, sealable rigid-walled container with 50-mL minimum volume. Use of a resealable plastic bags for holding and transporting the settled dust wipe sample is not recommended due to the potential losses of settled dust within the plastic bag during laboratory handling. Quantitative removal and processing of the settled dust wipe sample by the laboratory is significantly improved through the use of sealable rigid walled containers.

2.1.4 Sampling template, 1 ft^2 inside area reusable aluminum or plastic, or disposable cardboard or plastic template. A variety of shapes are recommended for use in variable field situations such as square, rectangular, square "U" shaped, rectangular "U" shaped, and "L." All templates must have accurately known inside dimensions. Templates should be thin (less than $^1/_8$ inch) and capable of lying flat on a flat surface.

2.1.5 Steel or plastic measuring tape or ruler, divisions to $^1/_{16}$ inch.

2.1.6 Wipe, disposable towelette moistened with a wetting agent. Wipe brands or sources should contain insignificant background lead levels. Laboratory analysis on replicate blank wipes should be used to determine background lead levels prior to use in the field. Background lead levels less than 10 μg per wipe are considered insignificant for most dust sampling activities. It is recommended to avoid brands of wipes that contain aloe because wipes containing aloe have been found to contain higher background lead levels. Increased laboratory sample preparation difficulties have also been noted for wipes containing lanolin. Wipe brands or sources selected for use should be of adequate width and thickness to perform the collection procedure. A thin wipe which is approximately 6 inches by 6 inches is recommended. Use of large, multiply or extra thick wipes can cause problems with laboratory analysis. Use of wipes with smaller dimensions may not be capable of holding settled dust contained within the sampling area.

2.2 General Supplies

2.2.1 Field notebooks, bound with individually numbered pages, see subsection 4.1.

2.2.2 Indelible ink marker, black or blue.

2.2.3 Ink pens, black or blue.

2.2.4 Packaging tape, used for sealing shipping containers.

2.2.5 Plastic bags, trash bags with ties.

2.2.6 Plastic gloves, powderless. Gloves with powder should not be used to avoid potential contamination of samples from powder material.

2.2.7 Preprinted field forms, preprinted with sufficient entry lines to address documentation needs presented in subsection 4.1

2.2.8 Shipping containers, cardboard or plastic for interim storage and shipment of sample collection containers.

3.0 Sampling Procedure

Two sampling procedures are presented. One is to accommodate collection of a settled dust sample in an unrestricted area such as a floor (Template Assisted Sampling Procedure). The other is to accommodate collection of a settled dust sample in a restricted area such as a window channel (Confined Area Sampling Procedure). The Confined Area Sampling Procedure should only be used when the Template Assisted Sampling Procedure can not be used due to sampling location constraints. The Confined Area Sampling Procedure assumes the operator can be orientated to a collection position where the sampling location's width is greater than its depth. It also assumes that the depth is no larger than the dimensions of a wipe. If this is not true, then the Template Assisted Sampling Procedure should be used.

3.1 Template Assisted Sampling Procedure

Following is a summary of this procedure:

1. Select a sampling location.
2. Mark the sampling location using a template.
3. Perform first wiping: Side-to-side, fold the wipe.
4. Perform second wiping: Top-to-bottom, fold the wipe.
5. Perform third wiping: Clean-up the corners, fold the wipe, and store the sample.

The detailed procedure is as follows:

3.1.1 Pull on a pair of clean, powderless, plastic gloves.

3.1.2 Carefully place a clean template on the surface in manner that minimizes disruption of settled dust at the sampling location. Either tape or place a heavy object on the outside edge of the template to prevent it from moving during sample collection. An alternative to using a template is to mark an outline of the sampling location using masking tape as described in subsection 3.2.2.

3.1.3 Discard any gloves used to mark the area in a trash bag and pull on a new pair of clean, powderless, plastic gloves.

3.1.4 At the beginning of a sampling period (or if a new bulk-packed container of wipes is opened), remove a minimum of the top 2 wipes from the container and wipe off gloved fingers with each wipe as they are removed. Use the next wipe from the container to collect the sample.

3.1.5 <u>First Wiping, Side-to-Side</u>: Hold one edge of the wipe between the thumb and forefinger, draping the wipe over the fingers of a gloved hand. Hold fingers together, hand flat, and wipe the selected surface area, starting at either corner furthest away from the operator (referred to as a far corner), using a slow side to side (left-to-right or right-to-left) sweeping motion. During wiping, apply pressure to the finger tips.

At the end of the first pass from one side to the other, turn the leading edge of the wipe (the portion of the wipe touching the surface) 180 degrees, pulling the wipe path slightly closer to the operator and make a second side-to-side pass in the reverse direction, slightly overlapping the first pass. (The 180 degree turn is used to assure that the wiping motion is always performed in the same direction on the wipe to maximize dust pickup.) Continue to cover the sampling area within the template, using the slightly overlapping side-to-side passes with the 180 degree turns at each edge until the close corner of the template is reached. Carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half with the sample side folded inside the fold.

3.1.6 Second Wiping, Top-to-Bottom: Using a clean side of the wipe, perform a second wiping over the sampling area within the template starting from a far corner in the same manner used for the first wiping, except use a top-to-bottom sweeping of the surface. When the close corner of the template is reached, carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half (again) with the sample from this second wiping folded inside the fold.

3.1.7 Third Wiping, Clean Corners: Using a clean side of the wipe, perform a third wiping around the perimeter of the sampling area within the template to pick up any dust remaining in the corners. Start from one edge of the template and use the same wiping technique as described above. When the perimeter has been wiped and the starting location reached, carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half one more time with the sample from this third wiping folded inside the fold.

3.1.8 Insert the folded wipe into a sample collection container. Using a tape measure, verify the internal dimensions of the sampling template used to collect the sample and label the sample collection container with sufficient information to uniquely identify the sample and the dimensions of the selected dust sampling area (with units such as inches). Discard any gloves in the trash bag. If the template is a reusable type, clean the template with several clean wipes.

3.2 Confined Area Sampling Procedure

Following is a summary of this procedure:

1. Select a sampling location.
2. Mark the sampling location using masking tape.
3. Perform first wiping: One direction, Side-to-side, fold the wipe.

4. Perform second wiping: One direction (reverse), Side-to-side, fold the wipe.
5. Perform third wiping: Clean-up the corners, fold the wipe, and store the sample.

The detailed procedure is as follows:

3.2.1 Pull on a pair of clean, powderless, plastic gloves.

3.2.2 Mark an outline of the sampling location using masking tape. Care should be taken to minimize any disruption of dust at the sampling location. For areas that are dirty or contain high dust levels, new tape may have to be applied more than once to get adhesion to the surface. Discard any soiled tape in a trash bag.

3.2.3 Discard any gloves used to mark the area in a trash bag and pull on a new pair of clean, powderless, plastic gloves.

3.2.4 At the beginning of a sampling period (or if a new bulk-packed container of wipes is opened), remove a minimum of the top 2 wipes from the container and wipe off gloved fingers with each wipe as they are removed. Use the next wipe from the container to collect the sample.

3.2.5 First Wiping, One Direction, Side-to-Side: Hold one edge of the wipe between the thumb and forefinger, draping the wipe over the fingers of a gloved hand. Hold fingers together, hand flat, and wipe the selected surface area, starting at either corner furthest away from the operator (referred to as a far corner), using a slow side to side (left-to-right or right-to-left) sweeping motion. During wiping, apply pressure to the finger tips. At the end of the first pass from one side to the other, carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half with the sample side folded inside the fold.

3.2.6 Second Wiping, One Direction, Side-to-Side: Using a clean side of the wipe, repeat step 3.2.5 using a wiping motion in the reverse direction.

3.2.7 Third Wiping, Clean Corners: Using a clean side of the wipe, perform a third wiping around the perimeter of the sampling area to collect any dust remaining in the corners. Start from the middle of one edge of the area and use the same wiping technique as described above. When the perimeter has been wiped and the starting location reached, carefully lift the leading dust line into the wipe using a slight rolling motion of the hand to capture the dust inside the wipe. Fold the wipe in half one more time with the sample from this third wiping folded inside the fold.

4.1.3 Documentation Required for Each Sample Collected:

- An individual and unique sample identifier and date of collection. This must be recorded on the sample container in addition to the field data records (notebook or form).
- Name of person collecting the sample and specific sampling location data from which the sample was removed.

4.2 QC Samples

4.2.1 Blank Samples. Blank samples should be periodically collected at random throughout the sampling day at each sampling site. Two types of blank samples should be collected: field blanks and QC blanks. Both these blanks are collected in the same manner; however, they are used for different purposes.

4.2.1.1 Field blanks. Field blank samples are used to identify any potential systematic lead contamination present in the wipe and during the handling of samples during field collection and laboratory analysis activities. Field blanks should be collected in the same manner as used to collect field samples with the exception that no surface is wiped. Each wipe designated as a field blank should be removed from the bulk pack, folded to match the field samples, and placed into a labeled sample collection container.

Each field blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., blanks can then be submitted in a blind manner to the laboratory). It is recommended that field blanks be collected at a frequency of 1 per 20 field samples. At a minimum, three should be collected at each sampling site for each new pack of bulk wipes used for sample collection (i.e., one near the beginning of the sampling period at the site, one in the middle, and one near the end). Field blank lead results should not exceed 20 µg/sample. Lead results above this value should trigger an investigation into the potential cause and resampling of samples associated with the field blank may have to be undertaken. Large blank lead values can often be sporadic and not systematic; therefore, blank correction of field sample results using field blank data is not recommended.

4.2.1.2 QC blanks. QC blank samples are used for preparation of blind reference material samples described in subsection 4.2.2. QC blanks should be collected in exactly the same manner as described

3.1.8 Insert the folded wipe into a sample collection container. Using a tape measure, measure the dimensions of the sampled area and label the sample collection container with sufficient information to uniquely identify the sample and the dimensions of the selected sampling area (with units such as inches). Discard any gloves in the trash bag.

4.0 Quality Control

Adherence to quality control (QC) procedures is an important part of field sample collection. QC procedures, including documentation requirements, field QC samples, reference material check samples, and contamination avoidance are presented in this section.

4.1 Documentation

All field data related to sample collection must be documented. A field notebook or sample log form can be used to record field collection data. It is recommended to utilize both types of documentation records (field notebooks and preprinted sample log forms) for assuring collection of all relevant field data. Field data entries on documentation records must adhere to the following requirements:

4.1.1 General Documentation Requirements:

- All entries must be made using ink.
- Each page (notebook or form) must include the name of the person making the entries and the date of entries found on the page.
- Any entry errors must be corrected by using only a single line through the incorrect entry (no scratch outs), and marked with the initials of the person making the correction and the date of correction.
- An initial page that correlates initials to a specific name must be generated and maintained with field data records to trace any initials used in notebooks and on data forms.

4.1.2 Specific Sampling Site Documentation Requirements:

- General sampling site description.
- Project or client name, address, and city/state location.
- Information as to what specific collection protocol was used.
- Information as to the use of interim storage and sample shipment mechanisms.

for field blanks. Each QC blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., reference materials prepared from the blanks can then be submitted in a blind manner to the laboratory). It is recommended that QC blanks be collected at a frequency of 1 per 20 field samples. At a minimum, two should be collected at each sampling site (an extra should be collected to assure sufficient QC blanks are available in case problems are experienced during preparation of blind reference material samples).

4.2.2 <u>Blind Reference Material Samples</u>. Reference materials should be periodically submitted to the laboratory for analysis as a check on adherence to proper laboratory sample preparation and instrumental analysis methods. Prepare a blind reference material by placing an accurately weighed portion (0.3000-1.0000 gram) of a reference material into a wipe (QC blank). The wipe should be folded to the same degree as the field samples. Place the reference material containing wipe inside a labeled sample collection container. Include a dummy sampling area on the label to disguise the identity of the blind reference material. The weight of reference material should be chosen to produce a blind reference material sample that will produce a lead level between 200 and 1000 µg/sample. It is recommended that the frequency of these QC samples, submitted to the laboratory for lead determinations, be at least 1 per 20 field samples. Reference materials from NIST[6], such as SRMs 2709, 2711, and 2704, are readily available and can be used for preparing blind reference materials. Other sources of materials with known lead levels, such as performance samples from the ELPAT[7] program, also may be used to prepare blind reference materials.

4.3 Contamination Avoidance

The following work practices should be followed to prevent cross-contamination of samples:

- Avoid disturbing and tracking dust from one location to another by:
  - identifying and clearly marking all sampling locations upon arrival at the sampling site,
  - avoiding walking through or over any of the marked sampling location areas, and
  - instructing field team members to pull on new disposable shoe covers upon each entry into the building.
- Change gloves frequently. Collection of each new sample must be conducted with a new pair of gloves.

- Clean sampling equipment and measuring tapes frequently with wipes.
- Inspect all sampling equipment for cleanliness prior to collection of each sample. Always clean suspect equipment if in doubt.
- Do not open sample collection containers until needed to collect each sample.
- When using bulk packed wipes, at each sampling location, discard the first two wipes pulled from wipe container.

5.0 Glossary

5.1 Digestion, Sample preparation process that solubilizes lead present in the sample. The digestion process produces an acidified, aqueous solution called the digestate. A lead determination is made on the digestate during an instrumental measurement process.

5.2 Field Blank, See subsection 4.2.1.

5.3 Field Data, Any information collected at the sampling site.

5.4 Field Sample, Physical material taken from the sampling site that are targeted for lead determination.

5.5 Reference Material, Material of known composition containing a known amount of lead. These materials have typically been subjected to a large amount of lead determinations to develop a lead result known to a high degree of confidence.

5.6 Sample Collection Container, Container for holding and transporting the samples from the field to the laboratory. The internal volume of the container must be sufficient to hold the entire collected sample.

5.7 Sampling Location, Specified area within a sampling site that is subjected to sample collection. Multiple sampling locations are commonly designated for a single sampling site. An example would be at the bottom of a specific slide in a specific playground area.

5.8 Sampling Site, Local geographical area that contains the sampling locations. A sampling site is generally limited to an area that can be easily covered on foot. An example would be John Smith's house at 3102 Nowhere Avenue, Detroit, MI.

5.9 Sample Preparation, Process used to ready a sample received from the field for lead determination using instrumental measurement methods. The process is dependent on the sample type and can include a large number of steps such as homogenization, drying, splitting, weighing, digestion, dilution to a final known volume, and filtering.

5.10 QC Blank, See subsection 4.2.2.

D. Protocol for Collection of Dust Samples for Lead Determination Using Vacuum Sampling

1.0 Introduction

This protocol provides for the collection of settled dust samples from surfaces using vacuum methods. The protocol is suitable for the collection of settled dust samples from both hard or smooth and highly textured surfaces, such as brickwork and rough concrete, and soft, fibrous surfaces, such as upholstery and carpeting.

Procedures presented in this protocol are intended to provide a method for collection of dust from surfaces that can not be sampled using wipe collection methods. In addition, these procedures are written to utilize equipment that is readily available and in common use for other environmental sampling applications (i.e., air particulate sample collection).

Due to the flow dynamics inherent in the vacuum method, results for vacuum dust samples are not likely to reflect the total dust contained within the sampling area. This protocol generally will have a collection bias toward smaller, less dense, dust particles. However, the protocol, if performed as written, will generate dust lead data that will be consistent and comparable between operators performing the method. This protocol can be used to produce samples for lead determination results in both loading ($\mu g/ft^2$) and concentration ($\mu g/g$). It is recommended, however, that it not be used for the generation of concentration results due to particle size collection bias and potential errors intrinsic to processing and handling preweighed filters (or entire filter cassettes), which are required to determine total collected sample weight. Even though it is not normally recommended, this protocol includes procedures for generation of total collected sample weight.

Other vacuum sampling methods that utilize less common equipment, such as cyclone sample collectors, may be useful for collection of settled dust, particularly with respect to generation of more quantitative dust lead concentration results.

2.0 Equipment and Supplies

2.1 Sampling Equipment

2.1.1 Air-sampling pump, A portable, battery-powered air pump that is capable of a flow rate of 2.5 L/min through a filter cassette equipped with the nozzle specified in subsection 2.1.2. Inlet of the pump must be fitted with a nipple to accept the tubing sized to fit tightly on the outlet side of a filter cassette.

2.1.2 Collection nozzle, A piece of stainless steel or carbon-impregnated plastic machined or molded on each end as follows: one machined or molded end to accept the tubing sized to fit tightly on the inlet side of a filter cassette; the other machined or molded to form a thin rectangular opening of $^{1}/_{2}$ inch by $^{3}/_{64}$ inch.

2.1.3 Disposable shoe covers (optional), See subsection 4.3.

2.1.4 Filter cassette, 37-mm filter cassette, preloaded with 0.8-µm, pore-size Mixed-Cellulose Ester Filters (MCEF) and backup support pad. If lead concentration results are to be determined and reported, then a special handling of these cassettes is required, as described in subsections 3.3.1 and 3.3.2.

2.1.5 Masking tape, used for holding down sampling templates and marking sampling locations.

2.1.6 Soap bubble air flow meter or calibrated rotameter, equipped with inlet and outlet fittings sized to fit tubing used to connect the filter cassette to the air-sampling pump.

2.1.7 Sampling templates, one ft^2 inside area reusable aluminum or plastic, or disposable cardboard or plastic template. A variety of shapes are recommended for use in variable field situations such as: square, rectangular, square "U" shaped, rectangular "U" shaped, and "L." All templates must have accurately known inside dimensions. Templates should be thin (less than $^{1}/_{8}$ inch), and be capable of lying flat on a flat surface.

2.1.8 Secondary sample collection container, resealable plastic bags for holding and transporting the filter cassettes.

2.1.9 Steel or plastic measuring tape or ruler, divisions to at least $^{1}/_{16}$ inch.

2.1.10 Tubing, plastic, flexible tubing sized to fit tightly on both the inlet and outlet of a filter cassette and the inlet of the air-sampling pump.

2.1.11 Wipe, disposable towelette moistened with a wetting agent. Wipe brands or sources should contain insignificant background lead levels. Laboratory analysis on replicate blank wipes should be used to determine background lead levels prior to use in the field. Background lead levels less than 10 µg per wipe are considered insignificant for most dust-sampling activities. It is recommended to avoid brands of wipes that contain aloe because wipes containing aloe have been found to contain higher background lead levels.

2.2 General Supplies

2.2.1 Field notebooks, bound with individually numbered pages, see subsection 4.1.

2.2.2 Indelible ink marker, black or blue.

2.2.3 Ink pens, black or blue.

2.2.4 Packaging tape, used for sealing shipping containers.

2.2.5 Plastic bags, trash bags with ties.

2.2.6 Plastic gloves, powderless. Gloves with powder should not be used to avoid potential contamination of samples from powder material.

2.2.7 Preprinted field forms, preprinted with sufficient entry lines to address documentation needs presented in subsection 4.1

2.2.8 Shipping containers, cardboard or plastic for interim storage and shipment of sample collection containers.

3.0 Sampling Procedure

Two types of sampling procedures are presented. The first, Loading Only Vacuum Collection, is intended for collection of dust for lead loading determinations ($\mu g/ft^2$) only. The second, Collection on Preweighed Media, is intended for collection of dust for both lead loading ($\mu g/ft^2$) and lead concentration ($\mu g/g$) determinations. The later type has two options that differ in the methods used for determining the total collected sample weight.

3.1 Calibration of Air-Sampling Pump

Regardless of the type of the sampling procedure used (see subsection 3.2 or 3.3), the air-sampling pump used for sample collection must be calibrated prior to sample collection for any given day. The procedure for air pump calibration is as follows:

3.1.1 Label a filter cassette with an ink marker to distinguish it as one used for pump calibration (and not to be confused with or used for collection of a field sample). Remove the inlet and outlet plugs and place them in a labeled, resealable plastic bag.

3.1.2 Attach the filter cassette to the air-sampling pump with a piece of flexible tubing. Attach a collection nozzle to the inlet side of the filter cassette using a short section of tubing (less than ½ inch).

3.1.3 Insert a soap bubble meter, calibrated rotameter, or other equivalent calibrated flow rate measuring device in-line between the air pump and the filter cassette equipped with a nozzle.

3.1.4 Turn on the air pump and adjust the flow rate of the air-sampling pump (if possible) to achieve an air flow between 2.5-2.8 L/min. Replace the air-sampling pump if this flow rate cannot be reached. Document the calibration in field data records (notebook or forms).

At the end of the sample collection day, the calibration must be verified. Perform the verification in the same manner as indicated in steps 3.1.1, 3.1.2, 3.1.3, and 3.1.4 above. Document the calibration verification in a field data records (notebook or forms). If the calibration verification fails to reproduce the minimum flow rate of 2.5 L/min, then all samples collected during the day are questionable and should be discarded.

3.2 Loading Only Vacuum Collection Procedure

The following procedure assumes that concentration results will not to be determined. In addition, it assumes that the air-sampling pump has been warmed up, and the calibration has been performed as described in subsection 3.1.

Following is a summary of this procedure:

1. Select a sampling location.
2. Mark the sampling location.
3. Perform first vacuuming: One direction, side-to-side.
4. Perform second vacuuming: One direction, top-to-bottom.
5. Perform third vacuuming: One direction, side-to-side, store the sample.

The detailed procedure is as follows:

3.2.1 Pull on a pair of clean, powderless, plastic gloves.

3.2.2 Mark the area to be sampled using one of the following two procedures:

3.2.2.1 Template Assisted Marking. Carefully place a clean template on the surface in manner that minimizes disturbance of settled dust at the location. Either tape or place a heavy object on the outside edge of the template to prevent it from moving during sample collection.

3.2.2.2 Manual Marking of Sampling Area. Mark an outline of the sampling location using masking tape. Care should be taken to minimize any disruption of dust within the sampling location. For areas that are dirty or contain high dust levels, new tape may have to be applied more than once to get adhesion to the surface. Discard any soiled tape in a trash bag.

3.2.3 Discard any gloves used to mark the area in a trash bag and pull on a new pair of clean, powderless, plastic gloves.

3.2.4 If not prelabeled from prefield processing, label a filter cassette with an ink marker. Remove the inlet and outlet plugs and place them into a labeled resealable plastic bag. Attach the outlet to the air-sampling pump with a piece of flexible tubing. Attach collection nozzle to the inlet side of the filter cassette using a short section of new tubing (less than ½ inch). Always use a new section of tubing for the inlet side of the filter cassette.

3.2.5 First Vacuuming: One Direction, Side-to-Side: With the air-sampling pump on, vacuum the selected sampling surface area, starting at either of the corners furthest from the operator (referred to as a far corner), using a slow side to side (left-to-right or right-to-left) sweeping motion while holding the collection nozzle at an angle of approximately 45° to the sampling surface. Avoid pressing down hard on the sampling surface during sample collection. Move the nozzle at a rate of approximately 2-4 inches per second. At the end of the first pass from one side to the other, carefully lift the collection nozzle and repeat the vacuuming sweep in the same direction as the first, using a slightly closer overlapping pass. Care must be taken to avoid overloading of the filter cassette. Repeat the procedure until the entire sampling area has been covered using the one-direction, side-to-side sweeping motions.

Overloading will result in decreased air flow and a reduction in sampling efficiency and increased sampling bias toward smaller, less dense particles. A drop of air flow of more than 10% is an indicator of overloading. If overloading of samples becomes evident, reduce the sampling area to prevent filter overloading or use multiple cassettes for collection within the same sampling area.

3.2.6 Second Vacuuming: One Direction, Top-to-Bottom: With the air-sampling pump on, vacuum the selected sampling surface area, starting at a far corner, using a slow top-to-bottom sweeping motion in the same manner as described in subsection 3.2.5. Repeat the procedure until the entire sampling area has been covered using the one-direction, top-to-bottom sweeping motions.

3.2.7 Third Vacuuming: One Direction, Side-to-Side: With the air-sampling pump on, vacuum the selected sampling surface area, starting at a far corner, using the slow, one-direction, side-to-side sweeping motion described in subsection 3.2.5. Repeat the procedure until the entire sampling area has been covered using the one-direction, side-to-side sweeping motions.

3.2.8 Remove the filter cassette from the inlet and outlet tubing sections, replace the cassette plugs, and place the sample into a labeled resealable plastic bag. Using a tape measure, measure the dimensions of the sampled area to within $^1/_{16}$ inch (or verify the dimensions of the template) and label the plastic bag containing the sample with sufficient information to uniquely identify the sample and the dimensions of the selected sampling area (with units such as inches). Also record this information on a preprinted data form or in a field notebook. Discard the used gloves in the trash bag.

3.3 Loading and Concentration Vacuum Collection Procedure

The collection procedure used for reporting both loading and concentration results is the same collection procedure as described in subsection 3.2 with two exceptions. First, a prefield and postfield, stabilization-weighing procedure is required to determine the total sample weigh collected. These procedures are described below. Second, if the Option 2 procedures listed below are used for weight determinations, care must be exerted during all handling of the sample cassettes to avoid inadvertent additions of weight to the filter cassettes. Option 2 always requires that the sample cassettes be handled with gloves and never with bare hands.

The overall collection procedure assumes that the air-sampling pump has been warmed up and the calibration has been performed as described in subsection 3.1. Following is a summary of the overall collection procedure:

1. Perform prefield stabilization and gravimetric procedures.
2. Select a sampling location.
3. Mark the sampling location.
4. Perform first vacuuming: One direction, side-to-side.
5. Perform second vacuuming: One direction, top-to-bottom.
6. Perform third vacuuming: One direction, side-to-side, store the sample.
7. Perform postfield stabilization and gravimetric procedures.

The two options available for determining a prefield and postfield sample media weight, preweighed filter and preweighed filter cassette, are presented below:

3.3.1 Prefield Stabilization and Gravimetric Procedure, Option 1—Preweighed Filter

This procedure suffers from the lack of quantitative transfer of all dust clinging to the cassette during postfield processing. Therefore, this option is considered somewhat more qualitative than Option 2. However, unlike Option 2, it is not susceptible to weight errors resulting from inadvertent touching or improper handling of the filter cassettes between pre- and postfield processing.

3.3.1.1 Prefield Procedure. The filter inside the cassette (not the backup support pad) must be weighed to constant weight prior to sample collection (prefield) at known temperature and humidity conditions (i.e., desiccated at room temperature). This can be performed for preloaded filter cassettes as follows:

a. Pull on a new pair of clean, powderless, plastic gloves.
b. Place a unique sample identifier on the outside of each cassette targeted for preweight generation using indelible ink and allow to dry.
c. Using a clean screwdriver, separate the cassette rings that hold the filter in place. Place the rings on a clean, dry area, such as a plastic bag or equivalent surface.
d. Using clean plastic tongs, lift the filter from the cassette and place it in a clean, dry, labeled beaker, watch glass, or other equivalent labeled container.
e. Place the container with filter into a desiccator and allow the filter to stabilize to a constant weight. Periodically weigh and record the filter on a clean balance to determine weight stability. (Record all weights to ±0.0001 g.) A constant weight for this protocol is one that does not change more than ±0.002 gram for repeated measurements (minimum of 2) taken over a minimum of a 24-hour period. Using clean plastic tongs, replace the filter back into the cassette, reassemble the cassette, reweigh the container, and record the empty container weight. The prefield filter weight is the difference between the container plus filter weight and the container-only filter weight.
f. Place the preweighed filter inside the sample cassette into a resealable plastic bag container for transport to the field.

3.3.1.2 Postfield Procedure. The filter and dust inside the cassette (not the backup support pad) must be weighed to constant weight prior to laboratory sample preparation (postfield) at the same known temperature and humidity conditions used for prefield processing. This can be performed as follows:

a. Pull on a new pair of clean, powderless, plastic gloves for each sample handled.

b. Place a unique sample identifier on the outside of a clean digestion vessel (usually a borosilicate glass beaker) using indelible ink and allow to dry. Tare the beaker (determine and record the weight) to ±0.0001 g.
c. Using a clean screwdriver, carefully separate the cassette rings that hold the filter in place while holding the cassette over the labeled beaker. Allow any dust contained inside the cassette to fall into the beaker. Using clean plastic tongs, carefully lift the filter from the cassette and drop it into the beaker. Carefully tap any visible dust clinging to the inside of the cassette into the beaker.
d. Place the beaker with filter into a desiccator and allow the filter to stabilize to a constant weight. Periodically weigh and record the weight of the container plus filter. (Record all weights to ±0.0001 g.) A constant weight for this protocol is one that does not change more than ±0.002 gram for repeated measurements (minimum of 2) taken over a minimum of a 24-hour period. The postfield filter weight is the difference between the container plus filter and dust weight and the container-only weight. Due to the potential of the dust to have significant water absorption, stabilization times for postfield weighing is expected to be considerably longer than for prefield gravimetric. It is recommended (not required) that no initial weight data be attempted until the sample has remained in the desiccator for at least 72 hours.
e. The entire sample plus filter in the beaker must be prepared for lead analysis. The total sample weight for use in determining lead concentration is the difference between the postfield filter weight and the prefield filter weight.

3.3.2 Prefield Stabilization and Gravimetric Procedure, Option 2—Preweighed Filter Cassette

This procedure results in a better quantitative transfer of all dust clinging to the cassette during postfield processing. Therefore, this option is considered somewhat more quantitative than Option 1. However, it is susceptible to weight errors resulting from inadvertent touching or improper handling of the filter cassettes between pre- and postfield processing.

3.3.2.1 Prefield Procedure. The entire filter cassette must be weighed to constant weight prior to sample collection (prefield) at known temperature and humidity conditions (i.e., desiccated at room temperature). All handling of the cassettes must be done with gloves. This can be performed for preloaded filter cassettes as follows:

a. Pull on a new pair of clean, powderless, plastic gloves.
b. Place a unique sample identifier on the outside of each cassette targeted for preweight generation using indelible ink and allow to dry.

c. Remove the inlet and outlet plugs and place them into a labeled, resealable plastic bag.

d. Place the filter cassette into a desiccator in a manner that allows air to flow freely through the inlet and outlet holes. Allow the filter cassette to stabilize to a constant weight. Record the weight of the entire filter cassette without plugs. (Record all weights to ±0.0001 g.) A constant weight for this protocol is one that does not change more than ±0.002 grams for repeated measurements (a minimum of 2) taken over a minimum of a 24-hour period.

e. Replace the inlet and outlet plugs and place the entire filter cassette with plugs into a labeled resealable plastic bag for transport to the field.

3.3.2.2 Postfield Procedure. The filter cassette with dust (without plugs) must be weighed to constant weight prior to laboratory sample preparation (postfield) at the same known temperature and humidity conditions used for prefield processing. This can be performed as follows:

a. Pull on a new pair of clean, powderless, plastic gloves.

b. Remove the inlet and outlet plugs and place them back into the original labeled, resealable plastic bag.

c. Place the filter cassette into a desiccator in a manner that allows air to flow freely through the inlet and outlet holes and that does not allow any spillage of dust out the holes. Allow the filter cassette to stabilize to a constant weight. Record the weight of the entire filter cassette plus dust without plugs. (Record all weights to ±0.0001 g.) A constant weight for this protocol is one that does not change more than ±0.002 grams for repeated measurements (minimum of 2) taken over a minimum of a 24-hour period. It is recommended (not required) that no initial weight data be attempted until the sample has remained in the desiccator for at least 72 hours.

d. The contents of the filter cassette should be prepared for lead analysis. A quantitative transfer procedure that utilizes the backup support pad for wiping dust out of the inside of the cassette combined with rinsing out the cassette with dilute acid can be used to transfer the entire sample to the digestion vessel. The total sample weight for use in determining lead concentration is the difference between the postfield filter cassette weight and the prefield filter cassette weight.

4.0 Quality Control

Adherence to quality control (QC) procedures is an important part of field sample collection. QC procedures, including documentation requirements, field QC samples, reference material check samples, and contamination avoidance are presented in this section.

4.1 Documentation

All field data related to sample collection must be documented. A field notebook or sample log form can be used to record field collection data. It is recommended to utilize both types of documentation records (field notebooks and preprinted sample log forms) for assuring collection of all relevant field data. Field data entries on documentation records must adhere to the following requirements:

4.1.1 General Documentation Requirements:

- All entries must be made using ink.
- Each page (notebook or form) must include the name of the person making the entries and the date of entries found on the page.
- Any entry errors must be corrected by using only a single line through the incorrect entry (no scratch outs) and marked with the initials of the person making the correction and the date of correction.
- An initial page that correlates initials to a specific name must be generated and maintained with field data records to trace any initials used in notebooks and on data forms.

4.1.2 Specific Sampling Site Documentation Requirements:

- General sampling site description.
- Project or client name, address, and city/state location.
- Information as to what specific collection protocol was used.
- Information as to the use of interim storage and sample shipment mechanisms.
- Prefield weight data including stabilization conditions for filter cassette.
- Postfield weight data including stabilization conditions for filter cassette.

4.1.3 Documentation Required for Each Sample Collected:

- An individual and unique sample identifier and date of collection. This must be recorded on the sample container in addition to the field data records (notebook or form).
- Name of person collecting the sample and specific sampling location data from which the sample was removed.

4.2 QC Samples

4.2.1 <u>Blank Samples</u>. Blank samples should be periodically collected (designated) throughout the sampling day at each sampling site. Two types of blank samples should be collected; field blanks and QC blanks.

Both these blanks are collected in the same manner; however, they are used for different purposes.

4.2.1.1 Field blanks. Field blank samples are used to identify any potential systematic lead contamination present in the filter cassette and handling of samples during field collection and laboratory analysis activities. Field blanks should be collected in the same manner used to collect field samples with the exception that no air is drawn through the filter cassette. Each cassette designated as a field blank should be removed from the plastic bag, inlet and outlet caps pulled off, the tubing and sampling nozzle attached, and then this procedure is reversed. The vacuum pump is not turned on.

Each field blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., blanks can then be submitted in a blind manner to the laboratory). It is recommended that field blanks be collected (or designated) at a frequency of 1 per 20 field samples. At a minimum, one field blank should be collected at each sampling site. Field blank lead results should not exceed 20 µg/sample. Lead results above this value should trigger an investigation into the potential cause of the problem and resampling of samples associated with the field blank may have to be undertaken. Large blank lead values can often be a sporadic and not systematic; therefore, blank correction of field sample results using field blank data is not recommended.

4.2.1.2 QC blanks. QC blank samples are used for preparation of blind reference material samples described in subsection 4.2.2. QC blanks should be collected in exactly the same manner as described for field blanks. Each QC blank must be labeled with its own unique identifier. The identifier for all blanks should be similar to other field samples to mask the identify of the blank from the laboratory (i.e., blind reference materials prepared from the blanks can then be submitted in a blind manner to the laboratory). It is recommended that QC blanks be collected (or designated) at a frequency of 1 per 20 field samples. At a minimum, two should be collected at each sampling site (an extra should be collected to assure sufficient QC blanks are available in case problems are experienced during preparation of blind reference material samples).

4.2.2 Blind Reference Material Samples. Reference materials should be periodically submitted to the laboratory for analysis as a check on adherence to proper laboratory sample preparation and instrumental analysis methods. Prepare a blind reference material by placing an accurately weighed portion (0.3000-1.0000 gram) of a reference material into a blank filter cassette (a collected QC blank). Place the cassette with weighed reference material inside a labeled, sample collection container. Include a dummy sampling area on the label to help disguise the blind reference material sample. The weight of reference material should be chosen to produce a blind reference material sample that will produce a lead level between 200 and 1000 µg/sample. It is recommended that the frequency of these QC samples submitted to the laboratory for lead determinations be at least 1 per 20 field samples. Reference materials from NIST[6], such as SRMs 2709, 2711, and 2704, are readily available and can be used for preparing blind reference materials. Other sources of materials with known lead levels, such as performance evaluation materials from the ELPAT[7] program, also may be used to prepare blind reference materials.

4.3 Contamination Avoidance

The following work practices should be followed to prevent cross-contamination of samples:

- Avoid disturbing and tracking dust from one location to another by:
  - identifying and clearly marking all sampling locations upon arrival at the sampling site,
  - avoiding walking through or over any of the marked sampling location areas, and
  - instructing field team members to pull on new disposable shoe covers upon each entry into the building.
- Use a new pair of powderless gloves at each sampling location.
- Inspect all sampling equipment for cleanliness prior to collection of each sample. Always clean suspect equipment if in doubt.
- Do not open sample collection containers until needed to collect each sample.
- When using bulk packed wipes, at each sampling location, discard the first two wipes pulled from the wipe container.

5.0 Glossary

5.1 <u>Digestion</u>, Sample preparation process that solubilizes lead present in the sample. The digestion process produces an acidified, aqueous solution called the digestate. A lead determination is made on the digestate during an instrumental measurement process.

5.2 <u>Field Blank</u>, See subsection 4.2.1.

5.3 Field Data, Any information collected at the sampling site.

5.4 Field Sample, Physical material taken from the sampling site that is targeted for lead determination.

5.5 Reference Material, Material of known composition containing a known amount of lead. These materials have typically been subjected to a large number of lead determinations to develop a lead result known to a high degree of confidence.

5.6 Sample Collection Container, Container for holding and transporting the samples from the field to the laboratory. The internal volume of the container must be sufficient to hold the entire collected sample.

5.7 Sampling Location, Specified area within a sampling site that is subjected to sample collection. Multiple sampling locations are commonly designated for a single sampling site. An example would be at the bottom of a specific slide in a specific playground area.

5.8 Sampling Site, Local geographical area that contains the sampling locations. A sampling site is generally limited to an area that can be easily covered on foot. An example would be John Smith's house at 3102 Nowhere Avenue, Detroit, MI.

5.9 Sample Preparation, Process used to ready a sample received from the field for lead determination using instrumental measurement methods. The process is dependent on the sample type and can include a large number of steps such as homogenization, drying, splitting, weighing, digestion, dilution to a final known volume, and filtering.

5.10 QC Blank, See subsection 4.2.2.

E. References

1 ASTM ES 29-94, Practice for the Field Collection of Soil Samples for Lead Determination by Atomic Spectrometry Techniques, American Society of Testing and Materials, Committee E-6, Performance of Buildings, ASTM PCN:03-506194-10, 1994.

2 ASTM ES 30-94, Practice for the Field Collection f Settled Dust Samples Using Wipe Sampling Methods for Lead Determination by Atomic Spectrometry Techniques, American Society of Testing and Materials, Committee E-6, Performance of Buildings, ASTM PCN:03-506194-10, 1994.

3 Environmental Protection Agency, Office of Solid Waste, Test Methods for Evaluating Solid Waste Physical/Chemical Methods, U.S. EPA SW 846, Third Edition, Proposed Update II.

4 ASTM ES 36-94, Practice for Hot Plate Digestion of Dust Samples for Determination of Lead by Atomic Spectrometry, American Society of Testing and Materials, Committee E-6, Performance of Buildings, ASTM PCN:03-506194-10, 1994.

5 ASTM E 1613-94, Test Method for Analysis for Lead by Inductively Coupled Plasma Atomic Emission Spectrometry (ICP-AES), Flame Atomic Absorption Spectrometry (FAAS), or Graphite Furnace Atomic Absorption Spectrometry (GFAAS) Technique, American Society of Testing and Materials, Committee E-6, Performance of Buildings ASTM PCP:03-506194-10, 1994.

6 National Institute of Standards and Technology, U.S. Department of Commerce, Technology Administration, Standard Reference Materials Catalog, 1993-94.

7 Environmental Lead Proficiency Analytical Testing (ELPAT) Program, performance samples remaining after performance testing rounds, ELPAT Coordinator, American Industrial Hygiene Association (AIHA), 2700 Prospect Avenue, Suite #250, Fairfax, VA 22031, (703) 849-8888.